

AUG 22 1922

DEPARTMENT OF COMMERCE.

BUREAU OF STANDARDS.

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CIRCULAR OF THE BUREAU OF STANDARDS.

No. 126.

(Issued July 27, 1922.)

UNITED STATES GOVERNMENT SPECIFICATION FOR
SALT WATER SOAP.

FEDERAL SPECIFICATIONS BOARD.

STANDARD SPECIFICATION No. 29.

This Specification was officially adopted by the Federal Specifications Board on June 20, 1922, for the use of the Departments and Independent Establishments of the Government in the purchase of materials covered by it.

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1. GENERAL.

The soap desired under this specification is a soap well made from pure coconut oil and the necessary alkali. It must be entirely soluble in both sea water and fresh water and make a suitable lather. Bidder shall state size, weight, and number of cakes in each box.

Failure to meet any of the following requirements will be cause for rejection:

Matter volatile at 105° C. shall not exceed 55 per cent.

Deliveries which yield more than 55 per cent of volatile matter will be rejected without further test.

Total matter insoluble in alcohol shall be not less than 2 per cent nor more than 3 per cent and shall consist, essentially, of sodium carbonate (Na_2CO_3).

Free alkali, calculated as sodium hydroxide (NaOH) shall not exceed 0.5 per cent.

Matter insoluble in water shall not exceed 0.5 per cent.

Chloride, calculated as sodium chloride (NaCl), shall be not less than 2.5 per cent nor more than 3.5 per cent.

Rosin, sugar, and foreign matter shall not be present.

The acid number of the mixed fatty acids prepared from the soap shall be not less than 250.

The percentage of matter volatile at 105°C . will be computed on the basis of the soap as received, but all other constituents will be calculated on the basis of material containing 55 per cent of volatile matter. The material will be purchased by net weight.

2. SAMPLING.

One cake shall be taken at random from not less than 1 per cent of the vendors' shipping containers, provided such containers contain not less than 50 pounds each. In the case of smaller containers a cake shall be taken at random from each lot of containers totaling not to exceed 5,000 pounds. The total sample shall in all cases consist of not less than three cakes taken at random from separate containers. With very large lots where the samples drawn as above will amount to more than 20 pounds the percentage of packages sampled shall be reduced, so that the amount drawn shall not exceed 20 pounds.

Wrap the individual cakes tightly in paraffined paper at once and seal by rubbing the edges with a heated iron. The inspector should accurately weigh each wrapped cake, record its weight and the date of weighing on the wrapper, place the wrapped cakes in an air-tight container, which should be nearly filled, seal, mark, and send to the laboratory for test. Samples should be kept cool until tested. The seller shall have the option of being represented at the time of sampling and when he so requests shall be furnished with a duplicate sample.

3. LABORATORY EXAMINATION.

(a) PREPARATION OF SAMPLE.—In case of samples that can be easily disintegrated and mixed, run the entire sample through a suitable chopper, except where the sample is large, when each

cake may be quartered and one-quarter of each cake run through the chopper. With samples that can not be handled as above, select a cake of average weight, quarter by cutting at right angles in the center, and shave equally from all freshly cut surfaces sufficient soap for analysis. Mix and weigh out all portions for analysis promptly. Preserve the remainder in an air-tight container in a cool place.

When a determination shows nonconformity with specification, a duplicate shall be run.

(b) **MATTER VOLATILE AT 105° C.**—Weigh 5 g of the sample in a porcelain or glass dish, about 6 to 7 cm in diameter and 4 cm deep, dry to constant weight in a vacuum oven or an inert atmosphere at a temperature not exceeding 105° C. (Time can be saved by having a layer of about 3 mm of ignited sand and a small stirring rod weighed with the dish and dissolving the sample in absolute alcohol, evaporating to dryness, breaking up sample with the rod, adding more alcohol, again evaporating and completing the drying in the oven as above.) Report loss in weight as matter volatile at 105° C.

(c) **TOTAL MATTER INSOLUBLE IN ALCOHOL, FREE ALKALI, OR FREE ACID.**—(1) *Matter Insoluble in Alcohol.*—Digest hot a 10 g sample with 200 cc of freshly boiled neutral ethyl alcohol (94 per cent or higher). Filter through a counterpoised filter paper neutral to phenolphthalein, or a weighed Gooch crucible with suction, protecting the solution during the operation from carbon dioxide and other acid fumes. Wash the residue on the paper or in the crucible with hot neutral alcohol until free from soap. Dry the filter paper or crucible and residue at 100° to 150° C. for three hours, cool, and weigh the total matter insoluble in alcohol.

(2) *Free Alkali or Free Acid.*—Titrate the filtrate from the above, using phenolphthalein as indicator, with standard acid or alkali solution, and calculate the alkalinity to sodium hydroxide (or potassium hydroxide) or acidity to oleic acid.

(3) *Matter Insoluble in Water.*—Proceed as in the determination of matter insoluble in alcohol. After filtering and thoroughly washing the residue extract it with water at 60° C. and wash the filter thoroughly. (When the matter insoluble in water is all inorganic, boiling water may be used for the extraction and washing.) Dry the filter and residue at 100° to 105° C. for three hours, cool, and weigh matter insoluble in water. The nature of this may be determined by further examination.

(4) *Total Alkalinity of Matter Insoluble in Alcohol (Alkaline Salts).*—Titrate the filtrate from the determination of matter insoluble in water with standard acid, using methyl orange as indicator. Calculate alkalinity to sodium carbonate (Na_2CO_3).

(d) *CHLORIDE.*—Dissolve 5 g of the sample in 300 cc of water, boiling if necessary to effect solution of all soluble matter. Add an excess of neutral chlorine-free magnesium nitrate solution (about 25 cc of a 20 per cent $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ solution). Without cooling or filtering titrate with standard silver nitrate solution, using potassium chromate as indicator. Calculate the chloride as sodium chloride.

(e) *ROSIN.*—A qualitative test for rosin may be made as follows: After decomposing a solution of the soap and separating the fatty acids heat a small quantity of the latter with acetic anhydride, cool, place a few drops on a spot plate, and add a drop of H_2SO_4 (specific gravity = 1.53). A fugitive violet color indicates the presence of rosin.

(f) *SUGAR.*—A qualitative test for sugar may be made as follows: Add a decided excess of hydrochloric acid to a solution of the soap, heat on a steam bath for 15 minutes, cool, filter from fatty acids, and test a portion of the filtrate which has been neutralized with sodium hydroxide solution by boiling for two minutes with an equal volume of boiling Fehling solution. The formation of red cuprous oxide indicates the presence of sugar.

(g) *ACID NUMBER.*—(1) *Preparation of Fatty Acids.*—Dissolve about 50 g of the soap in 300 cc of hot water, transfer to a separatory funnel, add 150 cc of approximately $2\text{NH}_2\text{SO}_4$, cool somewhat, add 120 cc of ether, shake, draw off the acid layer, and wash the ether layer free from acid with a strong salt (NaCl) solution. Then draw off the aqueous layer as completely as possible, transfer the ether layer to a flask (it is not necessary to transfer quantitatively), and 20 to 30 g of anhydrous sodium sulphate ($\text{Na}_2\text{S}_2\text{O}_4$), stopper the flask, shake, and let stand at a temperature below 25°C . until the ethereal liquid becomes perfectly clear, showing that all water has been taken up by the sodium sulphate. Filter through a dry paper into another Erlenmeyer flask and completely evaporate off the ether by passing through the flask a current of dry air, while heating the flask to a temperature not above 50°C .

(2) *Determination.*—In a 250 cc Erlenmeyer flask dissolve about 2 g of the fatty acids accurately weighed in 20 to 30 cc

of neutral 95 per cent ethyl alcohol. Titrate with standard alkali, using phenolphthalein as indicator. Calculate acid number (mg of KOH per g of fatty acids).

4. REAGENTS.

(a) STANDARD SODIUM HYDROXIDE SOLUTION.—0.25 N, or about 10 g sodium hydroxide dissolved in water and diluted to 1 liter. Standardized against Bureau of Standards' benzoic acid.

(b) STANDARD SULPHURIC ACID SOLUTION.—0.5 N, or about 25.8 g strong sulphuric acid (specific gravity=1.84) diluted to 1 liter. Standardized against standard sodium hydroxide solution (a).

(c) STANDARD SILVER NITRATE SOLUTION.—0.10 N, or about 17 g of silver nitrate dissolved in water and diluted to 1 liter. Standardized against chemically pure sodium chloride.

(d) SULPHURIC ACID (SPECIFIC GRAVITY=1.53).—Mix 62.5 cc of strong sulphuric acid (specific gravity=1.84) with 61.5 cc of water.

(e) STANDARD ALCOHOLIC SODIUM HYDROXIDE SOLUTION.—Same as (a), excepting that 95 per cent ethyl alcohol is used instead of water.

(f) POTASSIUM CHROMATE SOLUTION.—A 10 per cent solution of potassium chromate in water.

(g) FEHLING SOLUTION.—(1) *Copper Sulphate Solution*.—Dissolve 34.639 g of copper sulphate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) in water and dilute to 500 cc.

(2) *Alkaline Tartrate Solution*.—Dissolve 173 g of Rochelle salts ($\text{NaKC}_4\text{H}_4\text{O}_6 \cdot 4\text{H}_2\text{O}$) and 50 g of sodium hydroxide in water and dilute to 500 cc. Mix equal volumes of (1) and (2) immediately before use.

